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BONDING OF A PYROLYTIC GRAPHITE HYPERVELOCITY IMPACT TARGET

Final Report

by

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TOPICAL REPORT

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HYPERVELOCITY IMPACT TARGET

by

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prepared for

National Aeronautics and Space Administration

September 15, 1965

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SUMMARY

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This investigation involved a limited evaluation of two possible methods of joining annealed flat plates of pyrolytic graphite and the selection of a suitable method to fabricate a pyrolytic graphite hypervelocity impact target. A carbonaceous cement filled with powdered pyrolytic graphite was selected for bonding double shear specimens which were tested at room temperature in air and 1300°F in argon. The joint shear strengths obtained, averaged 100 psi and 120 psi at room temperature and 1300°F, respectively. A pyrolytic graphite target consisting of 15 individual plates was successfully bonded using the carbonaceous cement.

Author

INTRODUCTION

Waste-heat space radiators armored with pyrolytic graphite to prevent critical damage by impacting meteoroids have been shown to be theoretically lighter in weight than radiators armored with more conventional structural materials (ref. 1). Since pyrolytic graphite is generally available in thin sheets ($\leq 1/4''$), it is frequently necessary to use several plates in order to obtain thicknesses required for design applications or for hypervelocity impact test targets. Previous hypervelocity impact tests on pyrolytic graphite targets composed of a series of plates clamped together were inconclusive because the unbonded target laminations suppressed any definite characteristics of the pyrolytic graphite material (ref. 2). In this investigation, two processes applicable for joining pyrolytic graphite plates are evaluated. The main criterion for acceptance was to achieve a joint strength that was at least as strong as the shear strength of the pyrolytic graphite material in the basal or ab-plane. The evaluation of the joints was performed at 1300°F in argon and at room temperature in air. This report presents pertinent details of each of the two joining processes together with a comparison of the shear strengths of the joints achieved. The procedure used to join the pyrolytic graphite impact target is also described.

DESCRIPTION OF MATERIALS

Annealed Pyrolytic Graphite. - Sixteen pyrolytic graphite plates were supplied by the NASA-Lewis Research Center. These plates had been previously annealed at approximately 5800°F in vacuum. The plates were approximately $1/4'' \times 2-1/2'' \times 4''$. Some of the plates had a rough appearance and were etched on faces which correspond to the ab or basal plane. Small pyrolytic graphite flakes could easily be loosened from the etched faces.

Apparently the annealing cycle made these plates much softer and easier to cleave than the usual vapor-deposited pyrolytic graphite stock.

Carbonaceous Cement. - This cement consists of phenol formaldehyde^(A) binder and a filler made from Grafoil^(B) powder. Sheets of Grafoil were pulverized in a hammer mill to obtain a -30 mesh powder. The cement was prepared by adding 23 w/o filler to 77 w/o phenol formaldehyde and stirring until a homogeneous blend was produced.

Pyroceram 9606^(C). - This material is a devitrified glass/ceramic whose nominal composition is lithium aluminum silicate. In this evaluation, it was used as a -325 mesh powder or as thin discs.

JOINING PROCEDURES

Joining of Pyrolytic Graphite with Pyroceram 9606. - Pyroceram 9606 had been joined to ATJ graphite by melting Pyroceram glass in a conical depression in a graphite adherend using R.F. heating^(D). As the Pyroceram glass began to melt, a Pyroceram rod was pushed into the molten mass, pressure was applied to the end of the rod, the R.F. Heating was shut off and the sample was cooled to room temperature before exposure to air. The resulting joint exhibited reasonable adherence and the Pyroceram appeared to wet the graphite. Consequently, additional experiments were conducted to study the feasibility of joining pyrolytic graphite with Pyroceram.

Powdered Pyroceram 9606 was first sandwiched between pyrolytic graphite adherends and, subsequently, these were heated to 2800°F in argon with a contact pressure of 20 psi applied to the sample as the pyroceram melted. In these experiments, most of the Pyroceram was extruded out of the joint since the Pyroceram did not completely wet the pyrolytic graphite adherend. (The flatness of the pyrolytic graphite surface also made it difficult to keep the molten Pyroceram in place.) This portion of the investigation was discontinued since the geometry of the impact target did not permit variation of the joint configuration to accommodate the molten Pyroceram.

- (A) Bakelite BV-1845 Phenolic Varnish made by Bakelite Co., Div. of Union Carbide Corporation.
- (B) Product of High Temperature Materials, Inc., Lowell, Mass.
- (C) Product of Corning Glass Works, Corning, New York.
- (D) Unpublished experimental work by R.B. Greikila and E.K. Davis of Westinghouse Central Laboratories, Pittsburgh, Pennsylvania.

Joining Pyrolytic Graphite with the Carbonaceous Cement. - The cement, formulated as described earlier, was applied to both adherends with a spatula so that the mating surfaces were completely wetted by the phenol formaldehyde binder in the cement. After coating with cement, the parts were joined and slight contact pressure was applied. Excess cement that extruded from the joint was removed with a spatula. The pressure was maintained until the cement had undergone the curing cycle. Curing of the cement was accomplished by heating in air at a rate of 15° per hour to 260°F . When the cured assembly had cooled to room temperature, the pressure was released. The edge surfaces were abraded lightly with 400 grit silicon carbide paper to remove any excess cement. Subsequently, the cemented assembly was first carbonized by heating to 1600°F at a rate of 120°F/hr in argon. Then, the assembly was graphitized by continuing the heating to 3450°F at a rate of 300°F/hr and held at this peak temperature for six hours.

TESTING OF DOUBLE SHEAR SPECIMENS

Double shear test specimens were obtained by cementing $3/8'' \times 3/8'' \times 1/4''$ coupons of pyrolytic graphite with the carbonaceous cement as described earlier. The coupons were obtained by cutting one of 16 annealed pyrolytic graphite plates. Three of these pieces were cemented together producing a double shear specimen. The mating surfaces joined in these samples were parallel to basal planes of the tiles. In all, eight specimens were fabricated by the aforementioned process. Similar samples were fabricated from unannealed pyrolytic graphite plates and were processed with the NASA specimens as controls for the cementing process.

The samples were subjected to shear loading at room temperature and 1300°F in argon at a loading rate of $.02 \text{ in/min}$ in order to obtain a measure of the joint strengths achieved. The shear fixture, which is shown in Figure 1, was made from a high-strength graphite. These tests were conducted in an Instron Universal Testing Machine within a Brew High Temperature Furnace, as shown in Figure 2. The results of these tests are summarized in Table I.

TABLE I

JOINT SHEAR STRENGTH

	<u>Room Temperature</u>		<u>1300°F (Argon)</u>
Unannealed control samples #1	168 psi	#3	152 psi
#2	42 psi	#4	89 psi
Average	100 psi		121 psi
Annealed samples #1	10.7 psi	#5	18.6 psi
#2	10.2 psi	#6	10.3 psi
#3	9.5 psi	#7	12.7 psi
#4	10.9 psi	#8	30.4 psi
Average	10.4 psi		18.0 psi

The increased strength level of the joints in unannealed pyrolytic graphite is apparent. In the unannealed samples, shearing of the joints occurred in the cement-tile interface, while failure of the annealed samples occurred through a combination of tearing in the annealed pyrolytic graphite as well as separation at the cement-tile interface. Consequently, it was presumed that the joint shear strength was greater than the shear strength of the annealed pyrolytic graphite material. The level of the shear strength of the carbonaceous cement was also presumed to be represented by the tests on the unannealed material.

FABRICATION OF THE PYROLYTIC GRAPHITE HYPERVELOCITY IMPACT TARGET

The previous experiments indicated that bonding the annealed pyrolytic graphite with the pyrolytic graphite-filled carbonaceous cement was a practical and effective method to fabricate the pyrolytic graphite impact target. Accordingly, the impact target was fabricated from the fifteen remaining pyrolytic graphite plates using the same process described earlier in this report. Pressure was applied and was maintained on the cement assembly with C-clamps during the cure cycle only. Excess cured cement was removed from the target edges by hand-sanding with 400 grit silicon carbide paper. After the target was subjected to the carbonization and graphitization cycles, it was inspected for the visual quality of the joints and possible delaminations in the pyrolytic graphite plates. The finished target appeared sound and completely bonded except for a few small delaminations in one of the fifteen plates. Figure 3 shows the completed target.

CONCLUSIONS

It is apparent that the fabrication of pyrolytic graphite impact targets by joining flat plates with carbonaceous cement is feasible. The use of Pyroceram glass cement was not suitable for joining flat pyrolytic graphite plates. The shear strength of the carbonaceous cement joint is on the order of 100 psi.

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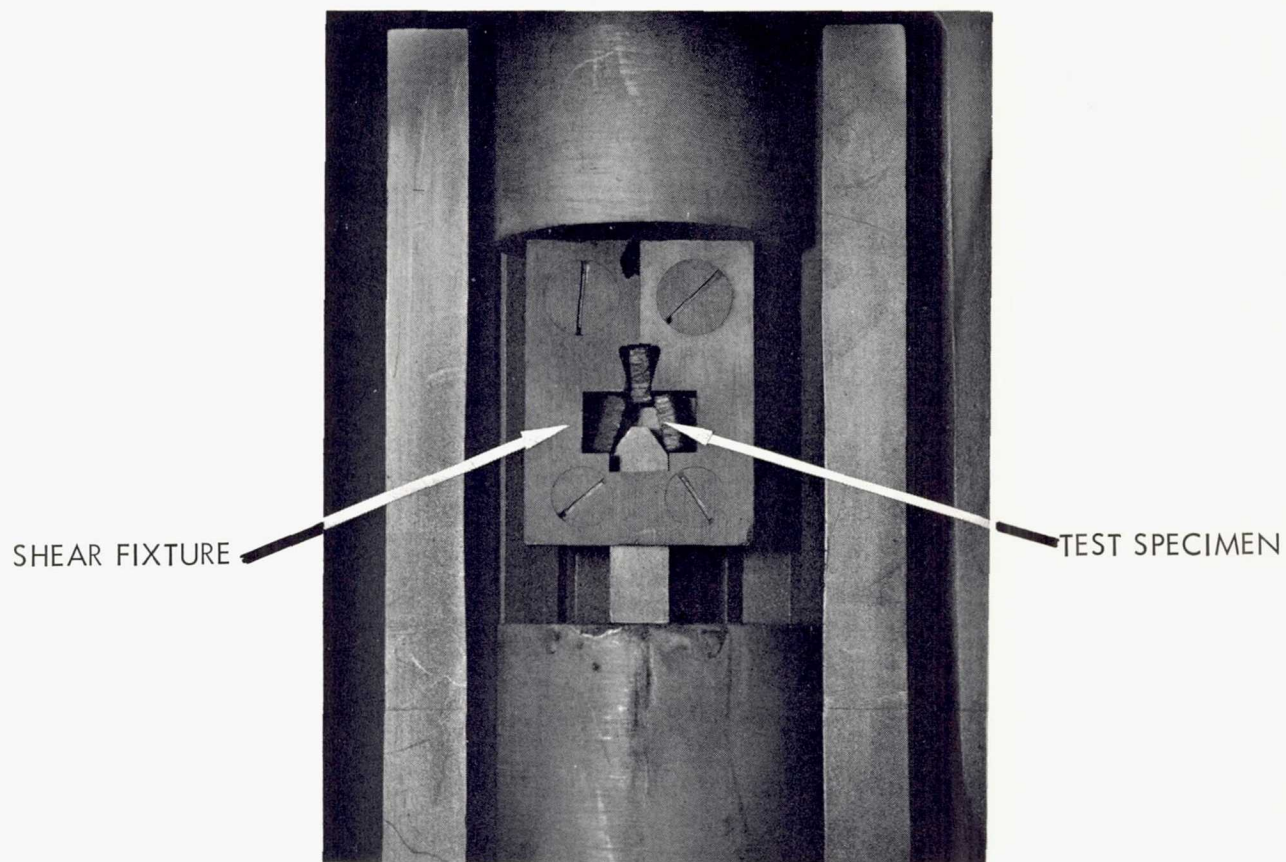


FIGURE 1 - GRAPHITE SHEAR FIXTURE
SHOWING COMPLETED TEST

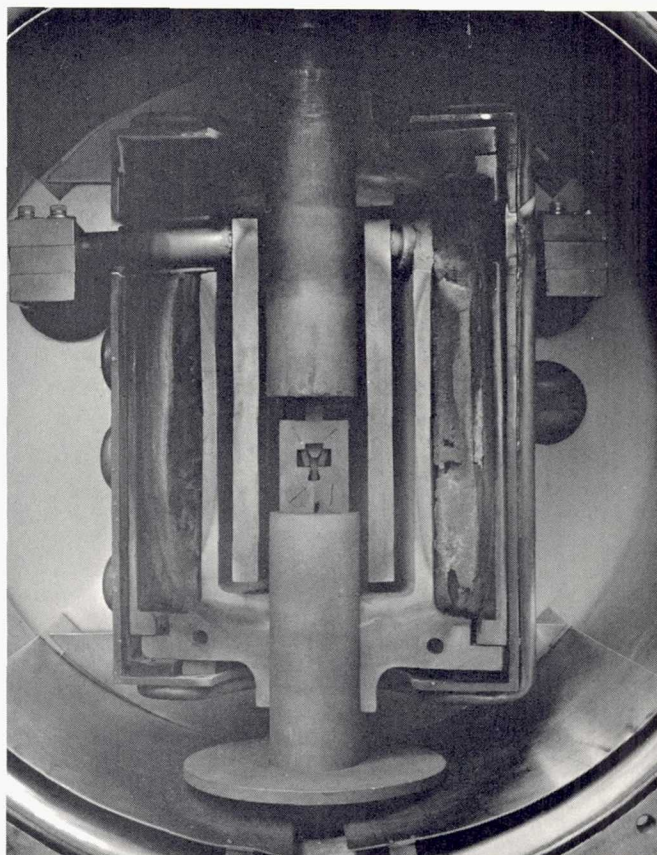


FIGURE 2 - VIEW SHOWING SHEAR FIXTURE WITHIN
THE BREW HIGH TEMPERATURE FURNACE

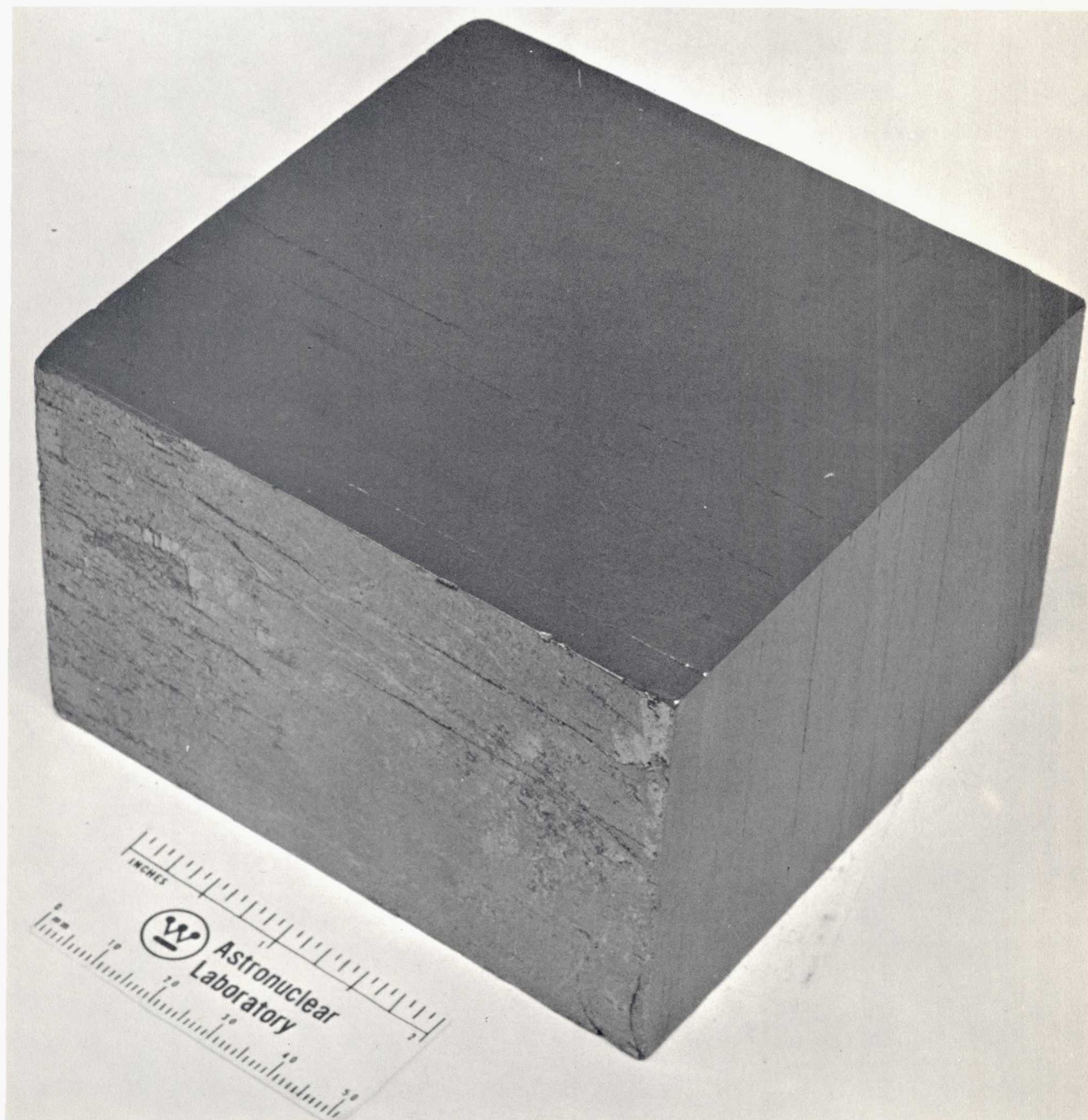


FIGURE 3 - VIEW OF THE BONDED PYROLYTIC GRAPHITE HYPERVELOCITY
IMPACT TARGET BLOCK COMPOSED OF 15 PLATES - 1/4"
THICK - FINAL HEAT TREATMENT - 3450°F

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